

# Tabulation of Apparatus Used for the Micro-Dumas Determination

Committee for the Standardization of Microchemical Apparatus, Division of Analytical Chemistry, AMERICAN CHEMICAL SOCIETY

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WITH the increase in the number of microchemical laboratories in recent years, and of the individual ideas probably existing in each, the committee felt that there might be a wide variety of microchemical apparatus used. Because no data were available on the extent of such variation, a questionnaire on the apparatus used in the Dumas determination of nitrogen was circulated in 1948, requesting such data as size of sample and description of apparatus parts. It also included a listing of those pieces of apparatus in use by the analyst which conformed to recommended specifications (2, 3). Sixty-four laboratories replied and in many instances reported more than one condition and type of equipment, or failed to reply to specific questions.

The committee believes that the information gained through this questionnaire is of general interest and has prepared the following summary of the data:

## 1. Sample Technique

- 53 used 0 to 5 mg.
- 34 used 6 to 10 mg.
- 16 used 11 to 50 mg.
- 26 used mixing tubes
  - 17 were 61 to 80 mm. long
  - 14 were 9 or more mm. in diameter

- 12 used a platinum boat
  - 6 were 11 to 15 mm. long
  - 9 were 3.6 to 5 mm. in width

- 43 used porcelain boats
  - 25 were 11 to 20 mm. in length

## 2. Carbon Dioxide Source

- 35 used dry ice
- 13 used marble
- 7 used calcite
- 3 used carbon dioxide from cylinders
- 6 used other sources

## 3. Carbon Dioxide Generators

### KIPP

- 17 used one Kipp generator
  - 13 were according to specifications (3)
- 3 used two Kipp generators in series
- 5 had a mercury trap

### DEWAR

- 18 used 1000-ml. capacity or less
- 10 used 1000 to 2000 ml.
- 4 used larger than 2000 ml.
- 20 reported charge lasting from 0 to 10 days
- 10 reported 11 to 20 days
- 2 reported longer than 20 days
- 29 used safety trap
- 5 did not use safety trap

### CARBON DIOXIDE FROM CYLINDERS

- 3 used this type

## 4. Gasometer

- 23 used gasometer
  - 19 were according to specifications (3)

## 5. Combustion Tube and Generator Connection

- 48 used a Z-tube of Pregl type

## 6. Combustion Tube

### TUBE DIMENSION

- 46 were according to specifications (3)

### TUBE COMPOSITION

- 18 used quartz
- 17 used Pyrex 172
- 29 used Vycor

### CONNECTION BETWEEN CARBON DIOXIDE SUPPLY TUBE AND COMBUSTION TUBE

- 50 used a rubber stopper

### IS THERE FREQUENCY OF BREAKAGE OF TIP OF COMBUSTION TUBE ON INSERTION?

- 8 reported yes
- 48 reported no

### SIZE OF RUBBER TUBING BETWEEN EXIT END OF COMBUSTION TUBE AND STOPCOCK OR NEEDLE VALVE

- 42 used tube of outside diameter 7.1 to 11 mm.
- 39 used tube of inside diameter 1.1 to 2 mm.
- 16 used 2.1 to 3 mm. inside diameter

### IS SHAPE OF TIP OF RECOMMENDED COMBUSTION TUBE (3) SATISFACTORY?

- 55 answered yes
- 2 answered no

### DIMENSIONS OF CAPILLARY TIP OF COMBUSTION TUBE

- Length
  - 15 were 26 to 30 mm.
  - 30 were 31 to 35 mm.

### Outside diameter

- 28 were 2.6 to 3 mm.
- 10 were 3.1 to 3.5 mm.

## 7. Stopcock or Needle Valve

### TYPE

- 53 used glass stopcock
  - 40 were according to specifications (2, 3)
- 4 used all-metal valve
- 2 used Hershberg-Southworth valve (1)

### CONNECTION OF NITROMETER TO STOPCOCK OR NEEDLE VALVE

- 1 used glass seal with cement
- 3 used  $\frac{1}{8}$  (standard taper) joints
- All others used rubber tubing having inside diameter of 1 to 3 mm., and outside diameter of 9 to 11 mm.

## 8. Nitrometer

- 35 used those according to specifications (3)
- 7 used their own design
- 43 used stem divisions of 0.01 ml.
- 45 used manufacturers' calibration values
- 4 used a water jacket
- 49 used a special clamp to hold reading lens
- 15 used a leveling bulb according to specifications (3)

## 9. Thermometer

- 61 used thermometers
  - 23 measured temperature in funnel
  - 34 measured temperature alongside stem
- 40 used thermometers calibrated in 0.1° C.
- 12 used thermometers calibrated in 0.25° C. or more

## 10. Long Furnace

There were 10 furnaces of individual design and 45 commercial

- 49 were electrically heated
- 24 used Nichrome wire
- 9 used Chromel wire
- 2 used platinum wire

- 13 furnaces were less than 18 cm. long
- 24 were 19 to 20 cm.
- 14 were 20 to 25 cm.
- 20 were rectangular
- 27 were round

- 20 were heated to 500° to 650° C.
- 9 were heated to 650° to 800° C.
- 9 were heated to less than 500° C.

- 28 furnaces were heated to 500° to 700° C. in region adjacent to sample
- 27 furnaces had average temperature of 500° to 700° C.

- 52 furnace temperatures were measured by means of thermocouple

- 21 measured temperature inside empty furnace
- 28 measured temperature inside empty combustion tube
- 12 of furnaces had built-in temperature measuring devices
- 2 furnaces had automatic temperature control

## 11. Sample Burner

- 42 used gas burners
- 19 used Bunsen burners
- 34 used gauze around combustion tube
- 18 used electric burners
- 10 were from 61 to 88 mm. long
- 6 were from 80 to 100 mm. long
- 12 had Nichrome wire elements

- 6 were heated to 600° to 700° C.
- 11 were heated to 700° to 800° C.
- 18 used a thermocouple
- 36 sample burners were operated manually
- 15 had motor-driven units
- 9 were home-made

- 14 burners were moved at constant rate
- 16 burners traveled 15 to 100 mm.
- 14 burners traveled 100 to 150 mm.
- 18 burners traveled distance in 11 to 20 minutes
- 11 burners required 21 to 30 minutes
- 4 burners required 41 to 50 minutes
- 1 burner required 50 to 60 minutes

- 13 burned sample once
- 35 burned sample twice

## 12. Copper Oxide

- 34 stored copper oxide in a glass bottle
- 52 used sieves
- 27 sieves were from 3 to 6 inches in diameter
- 27 used 21- to 40-mesh sieves
- 19 used 81- to 100-mesh sieves

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